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Mass Spectrometric Analysis and Characterization of Kepone in Environmental and Human Samples

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Mass Spectrometric Analyses and Characterization of Kepone in Environmental and Human Samples

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A specific portion of our environment has been contaminated with Kepone, or chlordecone. Additionally, some specific human exposures to high concentrations of Kepone have been confirmed. Gas chromatography mass spectrometry involving chemical ionization and high resolution mass spectrometry were used to detect, identify and confirm the presence of Kepone, Kepone photoproducts and a reduction product of Kepone in environmental and human samples. Field desorption, field ionization and electron impact mass spectrometric methods, as well as infrared and nuclear magnetic resonance techniques were used to characterize and identify Kepone hydrate and hemiketal in benzene and methanol solutions, respectively.

INTRODUCTION

Environmental contamination by Kepone[1,2] was discovered in 1975 in the vicinity of the sole United States producer, Life Science Products Company, Hopewell, Virginia. Since that time, Kepone has been under intense scrutiny from a methodology and toxicology viewpoint.

Kepone is a pesticide developed by the Allied Chemical Company in the early 1950s. Pure Kepone is a white powder and is highly effective in controlling banana and potato pests and is a potent ant and roach killer. It is used in about 55 commercial pesticide formulations but is predominantly used in ant and roach baits. Most of the Kepone produced has been exported for use on banana and potato pests.

The environmental samples collected at Hopewell, Virginia consisted of the following types: water and soil samples from the immediate area of the production plant; air samples within a 16 mile radius of the production plant; sediment, fish and shellfish samples from the James and Appomattox Rivers in the vicinity of Hopewell and down to Hampton Roads. Human samples were collected from employees of the manufacturing plant and from the general population. The samples were subjected to the specific clean-up procedures described by Moseman *et al.* and analyzed by GC electron capture.[1-4] Certain specific samples were then subjected to GC chemical ionization MS, GCMS and GC high resolution MS analyses for identification and confirmation of Kepone and transformation products. Mass spectrometric, NMR and IR analyses were performed on solutions of Kepone in benzene and methanol to determine its chemical reactivity and behavior in solution.

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EXPERIMENTAL

Instrumentation

Environmental samples were analyzed utilizing a Finnigan Model 3200 quadrupole mass spectrometer equipped with a CI source, Model 9500 gas chromatograph and Model 6100 data system. The experimental conditions were: ionization source temperature 60 °C; source pressure 950 microns; reagent and carrier gas, (ultra pure) methane, 40 ml min^{-1}; separator oven 220 °C and transfer lines 200 °C; injection port temperature 225 °C. The 183 cm × 2 mm i.d. glass GC column contained 3% OV-101 on 80/100 mesh Chromosorb W HP and was operated at 210 °C. The experimental retention time of Kepone was 252 s.

The EI mass spectrum of 'kepol',[5] found in the human feces sample, was obtained on a Hewlett-Packard 5930A mass spectrometer equipped with a model 5700A gas chromatograph. The range from *m/e* 150 to 650 was scanned at a rate of 100 *m/e* per second and the data were recorded on a light beam oscillograph, after manual observation of the mass spectrum on an oscilloscope. The feces sample was concentrated to 200 μl and 30 μl were injected, using a bypass over the membrane separator, before opening of the valve connecting the chromatograph spectrometer. Initial spectra were obtained by temperature programming from 80 °C (2 min) to 230 °C at 8 °C min^{-1}. Spectra and GC retention time of standard and sample were shown to be identical during isothermal runs made at 230 °C. A 180 cm × 2 mm i.d. glass column packed with 3% OV-17 on 100/120 mesh Gas-Chrom Q was used. The GC inlet temperature was 200 °C. The helium flow rate was 42 ml/min^{-1}. The transfer line to the mass spectrometer was maintained at 210 °C and the silicone membrane separator was kept at the same temperature as the GC oven.

Kepol, the Kepone hydrate and the hemiketal, were subjected to GC high resolution MS, FD, FI and EI mass spectrometric analyses utilizing a Varian MAT 311 A mass spectrometer equipped with a combination FDFIEI ion source. The mass spectrometer was interfaced to a Varian Model 2700 gas chromatograph and SS 100 data system and equipped with direct insertion probes.

The experimental GC high resolution MS conditions were: ion source temperature 240 °C; slit separator and connecting lines 250 °C; acceleration voltage 3 kV; electron energy 70 eV; electron current 2 mA; resolution 8000; perfluorokerosene (PFK) was used as a reference standard. The 183 cm×2 mm i.d. stainless steel GC column contained 3% Dexil 300 on 80/100 mesh Chromosorb W HP; helium flow rate of 15 ml min^{-1}; column temperature 210 °C; injection port temperature 255 °C.

Parameters used in FDFI analyses were: source temperature 60 °C; acceleration voltage 3 kV; extraction plate voltage 7 kV; FD emitter heating current 8–12 mA; resolution 1000–4000. The emitters were 10 micron diameter tungsten wire that had been activated in a benzonitrile partial pressure of about 10^{-3} Torr under the influence of high field strength and high temperature. The microneedle length was approximately 15–25 μm. The concentrations of the sample solutions were 1–20 $\mu g\,\mu l^{-1}$.

Parameters used in EI analysis were: source temperature 220 °C; acceleration voltage 3 kV; electron voltage 70 eV; electron current 1 mA; resolution 1000. Sample introduction was made via the direct insertion probe. The probe temperature was maintained at 20 °C. The concentrations of the sample solutions were 1–10 $\mu g\,\mu l.^{-1}$

NMR analyses were performed on a Varian XL-100 spectrometer equipped with a Nicolet Technology TT-100 Fourier Transform accessory. Operation was at 100.6 MHz for ^{1}H NMR analyses and at 25.14 MHz for ^{13}C NMR analyses.

Kepone, shown in Fig. 2, is 1,1a,3,3a,4,5,5,5a,5b,6-decachlorooctahydro-1,3,4-metheno-2*H*-cyclobuta-[*cd*]pentalen-2-one. Samples were supplied by the Allied Chemical Co., and were recrystallized from benzene, and shown to be 99% pure by GC.

'Kepol' (1,1a,3,3a,4,5,5,5a,5b,6-decachlorooctahydro-2-hydroxy-1,3,4-metheno-2*H*-cyclobuta[*cd*]-pentalene), the alcohol of Kepone, was prepared by the method of Gilbert *et al.*, by the reduction of Kepone with lithium aluminium hydride.[7]

'Monohydrokepone' is the 1a,3,3a,4,5,5,5a,5b,6-nonachlorooctahydro-1,3,4-methano-2*H*-cyclobuta-[*cd*]pentalen-2-one isomer shown in Fig. 3(a). 'Dihydrokepone' is one of two isomers, either 1a,3,4,5,5,5a,5b,6-octachloro-1,3,4-metheno-2*H*-cyclobuta[*cd*]pentalen-2-one, or 1a,3,3a,4,5,5,5a,6-octachloro-1,3,4-metheno-2*H*-cyclobuta-[*cd*]pentalen-2-one shown in Fig. 4(a). The monohydro and dihydro derivatives of Kepone were prepared in a manner similar to that of Alley *et al.*[8] The structure of the monohydrokepone and the two structures of the dihydrokepone were initially proposed by Alley, on the basis of their mass spectra, NMR spectra and chemical interconversions to Mirex photoproducts.[8] Mass spectral fragments were identical to those reported by Alley, and in addition, the structural assignments were confirmed and extended by a ^{1}H and ^{13}C NMR study carried out in our laboratory.[6]

Kepone (1.083 g) was dissolved in 40 ml of cyclohexane (Burdick and Jackson Laboratories, Inc., Muskegon, Michigan) in an Ace Glass photochemical reactor, equipped with a reflux condenser and magnetic stirrer, and irradiated for 2.5 h with a Hanovia 450 watt medium pressure mercury arc lamp. The photo-reactor vessel and quartz immersion well were water cooled.

The solution was concentrated on a rotary evaporator and sublimed at 150 °C, 0.01 Torr. The sublimate was dissolved in benzene and a portion was subjected to preparative GC on a Varian Aerograph 1520 Preparative Gas Chromatograph equipped with a 161 cm× 0.80 cm i.d. coiled stainless steel column (4% SE-30/6% OV-210 on Gas Chrom Q) and a thermal conductivity detector. Instrumental conditions were as follows: column temperature, 240 °C; injector temperature, 235 °C; detector temperature, 255 °C, thermal conductivity detector current, 150 mA; flow rate 40 ml min^{-1} helium. Under these conditions the dihydrokepone photoproduct eluted after approximately 26.5 min; the monohydro photoproduct, 36 min; and Kepone, 43 min.

The fraction containing the dihydrokepone was shown by analytical GLC (3% OV-1 on chromosorb W HP 80/100, 200 °C) to contain at least four minor components in addition to the major peak. The composition of the major peak was established by MS. Based on relative peak height measurements, the major peak constituted approximately 80% of the preparative GC fraction. Similarly, the fraction enriched in the monohydro photoproduct (93%) contained at least three minor components in addition to the major peak. The major component was shown to be monohydrokepone by MS.

RESULTS AND DISCUSSION

Selected environmental samples were subjected to GC chemical ionization MS analyses for Kepone, utilizing the selected ion monitoring technique to monitor the four largest ions—*m/e* 488.7, 490.7, 492.7 and 494.7—in the quasimolecular ion ([M+H]) region, Fig. 1. Nominal resolution was used with correction of the peak centroid for the instrumental offset and chlorine mass defect. This technique enhanced the instrument sensitivity and provided the isotope ratios for confirmational purposes. The minimum detectable level obtained from a standard solution of Kepone was approximately 700 pg. The chemical structure of Kepone (in the dehydrated, carbonyl form) and CI mass spectrum are shown in Fig. 2. The presence of Kepone was confirmed in many environmental and human samples (Table 1). The criteria used for confirmation of Kepone were GC retention time combined with quasimolecular ion ([M+H]$^{+}$), chlorine isotope ratios.

In addition to Kepone, a known photoproduct of Kepone was recognized in environmental samples[9] in this study. Monohydrokepone, $C_{10}HCl_9O$ was found in soil and several water samples from the immediate vicinity of the manufacturing site. This was confirmed by comparing the CI mass spectra of the molecular ion

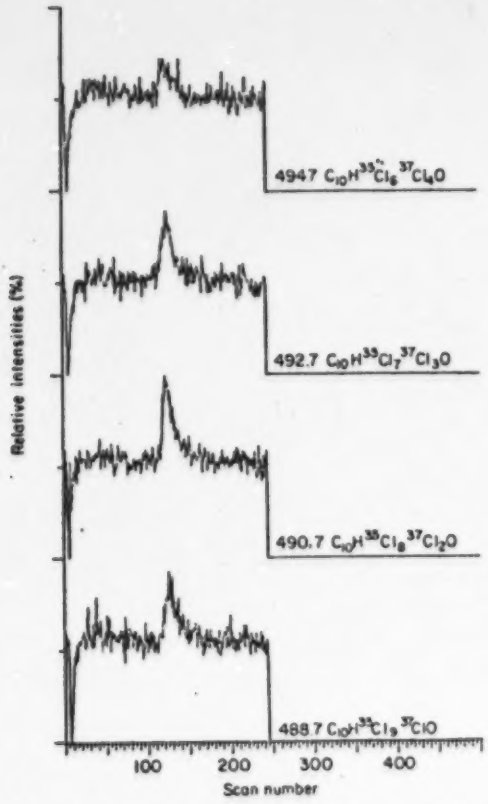


Figure 1. The GCCIMS selected ion monitor trace of a Kepone standard (3 ng).

Table 1. Confirmed presence of Kepone in environmental and human samples by GC chemical ionization MS

Type of sample	Concentration range[1]	Number of samples
Water	0.15 – 3.38 ppm	3
Air	0.21 – 54.8 $\mu g\ m^{-3}$	18
Soil, sediment, sludge	1.31 – 13,250 ppm	4 3 1
Fish with fins	1.04 – 14.4 ppm	10
Shellfish	0.18 – 0.91 ppm	10
Human sebum	0.05–3.06 μg per forehead wipe	10
Human blood*	2–15 ppm	2
Human feces*	2–5 ppm	2
Human bile*	0.1–1.5 ppm	3

* Unpublished data.

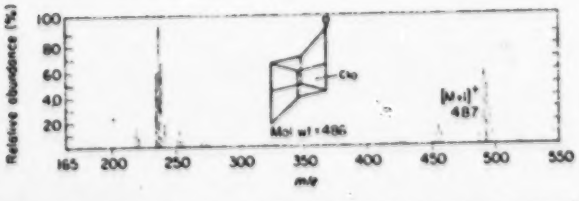


Figure 2. The structure and CI (methane) mass spectrum of Kepone.

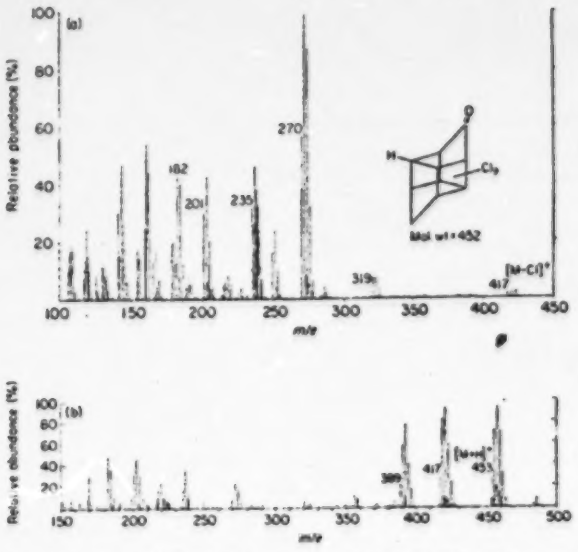


Figure 3. The chemical structure and EI (a) and CH_4 CI (b) mass spectra of monohydrokepone.

region and the GC retention time with that of the synthesized compound. The assigned chemical structure, EI and CI mass spectra are shown, respectively, in Fig. 3.

Figure 4 shows the EI and CI spectra obtained for dihydrokepone standard. One particular soil sample

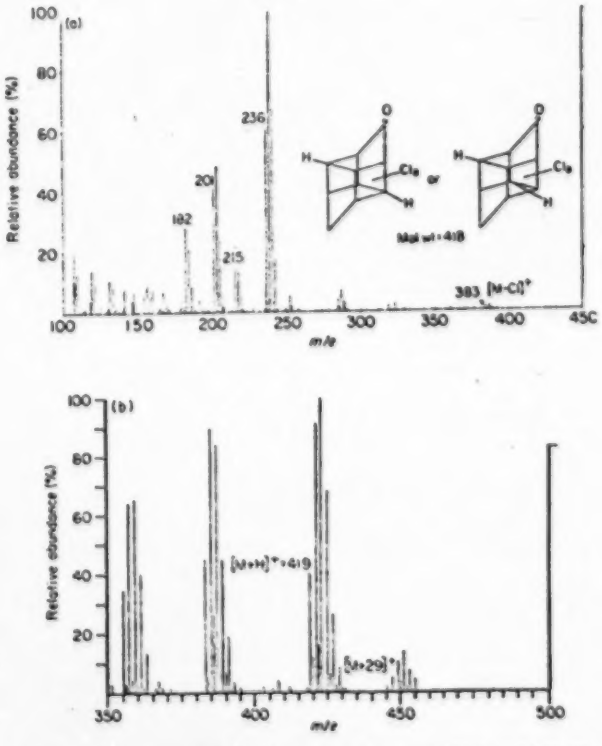


Figure 4. The possible chemical structures and EI (a) and CH_4 CI (b) mass spectra of dihydrokepone.

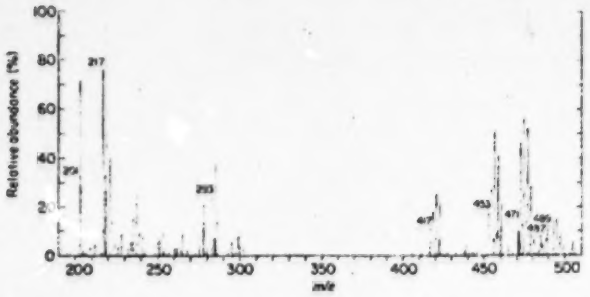


Figure 5. The experimental CI mass spectrum of the Kepone reduction product 'kepol' obtained from a human stool extract.

showed an electron capture GC peak at the same retention time as dihydrokepone, but there was not sufficient material present to allow for confirmation by GCMS.

In addition to known photoproducts found in the environment, a methane GC chemical ionization MS examination of a human feces extract from one of the workers in the Hopewell, Virginia, Life Science plant, provided the first encounter with a new conversion product of Kepone. Figure 5 shows the experimental CI spectrum of this compound which eluted from the GC column at approximately 2.5 times the retention time of Kepone. Assignment of the molecular ion was initially complicated by the apparent presence of $[M+H]^+$ and $[M-H]^+$ ions at *m/e* 489 and 487, respectively, and the small relative intensity of the first member of a ten chlorine isotope cluster. However, a GCMS run using EI ionization readily detected a nine chlorine isotope cluster at *m/e* 453 corresponding to the $[M-Cl]^+$ fragment of a molecular ion at *m/e* 488. The weaker chlorocluster corresponding to $[M]^{\ddagger}$ was also observed. The mass shift of two from Kepone suggested reduction of the carbonyl to a secondary alcohol, now called 'kepol'. The EI mass spectrum of a synthetic standard is shown in Fig. 6.

Fragments in the EI mass spectrum strongly supported this assignment. The base peak fragment at *m/e* 218 corresponded to the splitting of the molecule into an intense ion consistent with $C_5H_2OCl_4$, which was assigned to the tetrachlorocyclopentadienol radical cation (*a*), shown below. Related fragment ions assigned as *b* and *c*, were also present.

Such fragments were in direct analogy to the formation of chlorocyclopentadiene, or chlorocyclopentadieneone fragments for Mirex, Kepone and structurally related compounds. As for example, the assigned fragments, shown below: *d* and *e* from Mirex; *d*, *e* and *g* from Kepone; *c*, *d*, *e*, *f*, *g* and *h* from monohydrokepone; and *c*, *f* and *h* from dihydrokepone.[8,10,11]

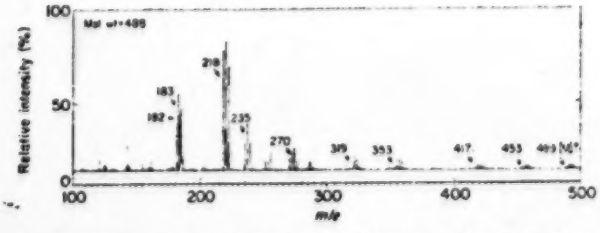
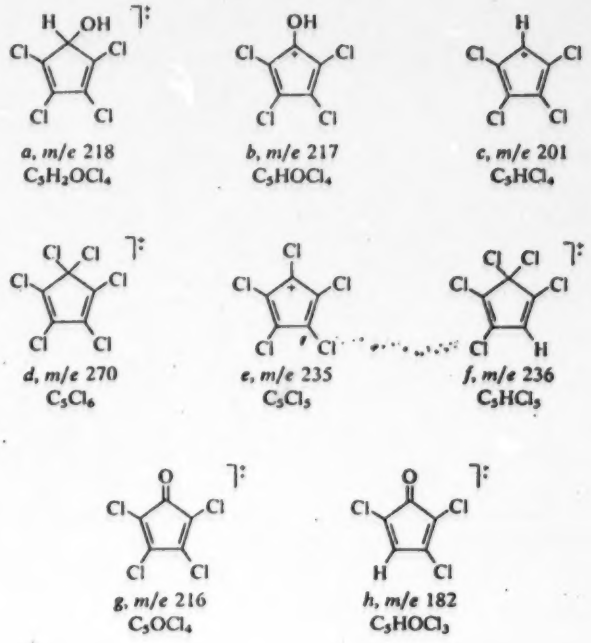


Figure 6. The EI mass spectrum of a synthetic standard of 'kepol', the alcohol formed by reduction of Kepone.



Fragments *d* and *e* of Scheme 1 were also found in the EI mass spectrum of the reduction product, as was a seven chlorine isotope cluster at *m/e* 353, probably corresponding to $[M-(Cl+2HCl+CO)]^+$, or $[M-(Cl_2+HCl+HCO)]^+$, or equivalent combined neutral losses.

In the experimental CI spectrum in Fig. 5, the fragment at *m/e* 471 corresponded formally to $[M-HO\cdot]^+$ or, more probably, to the $[(M+H)-H_2O]^+$ ion, a very reasonable loss for this cyclic secondary alcohol. The *m/e* 453 ($[M-Cl]^+$) cluster was observed, as well as one at *m/e* 417 which corresponded formally to $[M-(Cl+HCl)]^+$ and completed the fragments in the high mass region. The low mass region had an intense fragment ion corresponding to *b*, probably resulting from hydride abstraction from $[M]^{\ddagger}$ and subsequent fragmentation, as well as fragments corresponding to *a* and *c*.

The methane CI mass spectra of Kepone, monohydrokepone and dihydrokepone, shown in Fig. 2, Fig. 3(b) and Fig. 4(b), respectively, were each characterized by $[M+H]^+$, $[M-Cl]^+$ and $[M-(Cl+CO)]^+$ ions. In some cases, the weaker alkyl attachment ions, $[M+C_2H_5]^+$ and $[M+C_3H_5]^+$ could also be identified.

After low resolution GCMS examination, the human feces sample was subjected to GC high resolution MS analysis in order to rigorously establish the elemental composition of the GC peak that eluted substantially later than Kepone. The elemental compositions of the two masses, *m/e* 490 and *m/e* 492, were determined utilizing the GC high resolution MS peak matching technique. The values obtained were within 1.5 ppm of the theoretical values of 489.6961 and 491.6932 for $C_{10}H_2{}^{35}Cl_9{}^{37}ClO$ and $C_{10}H_2{}^{35}Cl_8{}^{37}Cl_2O$, respectively. These values, along with the low resolution EI analysis, GC retention time and comparison of data from the synthetic compound established the presence of 'kepol'.

The presence of kepol in a stool sample was initially

ascribed to metabolic reduction of the carbonyl, or more accurately, the *gem*-diol functionally, in order to produce an alcohol for possible conjugation and excretion. However, recent studies have indicated that solutions of Kepone and methyl alcohol can yield kepol as a reduction product.[12] Because the human stool sample was in contact with benzene + methanol mixture commonly employed in these analyses, the presence of kepol may be a product of this reduction. Thus, kepol may represent a previously unrecognized artifact in Kepone analysis.

GCMS analysis of Kepone in solution revealed only Kepone and the solvent in the mass spectra. Infrared and NMR analyses of Kepone in solution indicated that it existed as a mixture of the hydrate, $C_{10}H_2Cl_{10}O_2$ (Mol. wt. = 504) and nonhydrated Kepone in benzene and as the hemiketal, $C_{11}H_4Cl_{10}O_2$ (Mol. wt. = 518) in methanol. Direct insertion probe mass spectrometric techniques, FD, FI, and EI, confirmed the presence of the hydrate and hemiketal in solution.

Several samples of Kepone, analytical standard grade, in benzene were analyzed by ^{13}C and ^{1}H NMR. These contained varying amounts of water, both bound as the hydrate (*gem*-diol) and associated with the hydrate. In no case, could all of the water be removed from the samples. Samples sublimed under high vacuum and vacuum distilled into benzene were approximately 50% in the *gem*-diol form and 50% in the free carbonyl form. In acetonitrile, only the hydrate was observed, and in methanol, only the hemiketal. Further details will be published at a later date.[6]

The FDMS analyses were performed on 2 $\mu g\,\mu l^{-1}$ solutions of Kepone in pesticide quality benzene and methanol. The emitter heating current was rapidly adjusted to 12 mA while scanning between *m/e* 435 and *m/e* 555 to produce the intense molecular ion cluster shown in Fig. 7(a) and (b) for benzene and methanol, respectively. A slow adjustment of emitter heating current produces the same spectra but of lower intensity. Insignificant amounts of fragmentation occur between *m/e* 100 and *m/e* 486. The resolution was adjusted to 4000 in the FI mode and the peak matcher was calibrated with known compounds. The solutions were concentrated to about 10 $\mu g\,\mu l^{-1}$ and the two reference compounds, Kepone and Mirex, were added to each solution. The elemental compositions of *m/e* 504 and 506 in the Kepone hydrate and *m/e* 518 and 520 in the Kepone hemiketal were determined. The values obtained were within 10 ppm of the theoretical values, 503.6940, 505.6911, 517.7097 and 519.7067.

A mixture of the hydrate and hemiketal were subjected to FIMS analysis utilizing the direct insertion probe for sample introduction. The ion source temperature was maintained at 60 °C, the probe temperature was programmed from 25 to 200 °C while obtaining spectra. Kepone corresponding to the molecular weight of the dehydrated carbonyl form (Mol. wt = 486), was the predominant component in all spectra. Minor amounts of hydrate and hemiketal were evident in spectra obtained between 25 °C and 125 °C. The hydrate *m/e* 504 and hemiketal *m/e* 518 were predominant in the FD analyses performed on this mixture and suggest that the heat required for vaporization of these compounds is sufficient to cause dehydration to the carbonyl form.

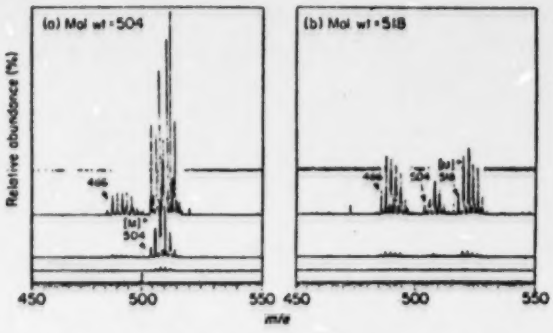


Figure 7. (a) The FD mass spectrum of Kepone hydrate. (b) The FD mass spectrum of Kepone hemiketal.

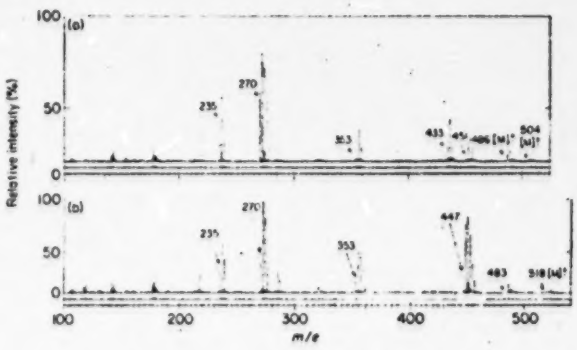


Figure 8. (a) The EI mass spectrum of Kepone hydrate. (b) The EI mass spectrum of Kepone hemiketal.

The EIMS analyses were performed on the hydrate and hemiketal utilizing the direct insertion probe for sample introduction. The ion source temperature was 220 °C and the direct probe temperature was maintained at 20 °C providing flash evaporation and/or sublimation of sample into the ion source. Spectra were obtained immediately after introduction of the probe. The weak molecular ions of Kepone and the hydrate were present in the EI analysis performed on the benzene solution, Fig. 8(a), and were in agreement with NMR analysis. The EI analysis performed on the solution of Kepone in methanol, Fig. 8(b), also agreed with NMR analysis in which only the hemiketal was detected. The EI, FD, FI and GCMS analyses of these two compounds indicated that decomposition of the hydrate and hemiketal forms of Kepone to the carbonyl form did occur in the GC system in GCMS analyses. Therefore, it is important that sufficient heat be applied to the GC injection port for these analyses.

In conclusion, GCMS analyses were instrumental in confirming that the environment within the Hopewell, Virginia area was contaminated by Kepone. Specific cases of human exposure to Kepone were also confirmed.

A Kepone photoproduct, a reduction product, the hydrate and the hemiketal were detected, confirmed and characterized utilizing various mass spectrometric, IR, NMR and chromatographic techniques.

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REFERENCES

1. Preliminary Report of Kepone Levels Found in Environmental Samples from Hopewell, Virginia Area—16 December 1975, Health Effects Research Laboratory, United States Environmental Protection Agency, Research Triangle Park, North Carolina 27711.
2. Preliminary Report on Kepone Levels Found in Human Blood from the General Population of Hopewell, Virginia—3 March 1976, Health Effects Research Laboratory, United States Environmental Protection Agency, Research Triangle Park, North Carolina 27711.
3. R. F. Moseman, H. L. Crist, T. R. Edgerton and M. K. Ward, *Arch. Environ. Contam. Toxicol.* in press.
4. Determination of Kepone in Shellfish, Fish, Feeds, Dairy Products, Eggs, Sediment, and Soils—17 February 1976, Commonwealth of Virginia, Division of Consolidated Laboratory Services, 1 North 14th Street, Richmond, Virginia 23219.
5. W. L. Dilling and M. L. Dilling, *Tetrahedron* 23, 1225 (1967).
6. N. K. Wilson and R. D. Zehr, presented at the Symposium on Fourier Transform NMR of the Twenty-Ninth Annual Southeastern Regional Meeting of the American Chemical Society, Tampa, Florida, 4 November 1977.
7. E. E. Gilbert, P. Lombardo, E. J. Rumanowski and G. L. Walker, *J. Agric. Food Chem.* 14, 111 (1966).
8. E. G. Alley, B. R. Layton and J. P. Minyard Jr., *J. Agric. Food Chem.* 22, 442, (1974).
9. Gas Chromatographic and Mass Spectral Behavior of Kepone and Three Related Compounds, by A. Borsetti and J. A. G. Roach, Division of Chemistry and Physics, and N. Hardin, District Laboratory, The Food and Drug Administration.
10. D. A. Carlson, K. D. Konyha, W. B. Wheeler, G. P. Marshall and R. G. Zaylskie, *Science* 194, 939 (1976).
11. S. Safe and O. Hutzinger, *Mass Spectrometry of Pesticides and Pollutants*, p. 128 CRC Press, Cleveland, Ohio (1973).
12. L. Wright, private communication.

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